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DETECTION OF CYCLOHEXANONE IN THE
ATMOSPHERE ABOVE EMPLACED ANTITANK
MINES

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PREFACE

This report was prepared by T.F. Jenkins, Research Chemist, and First Lieutenant W.F. O'Reilly, Chemist, under the supervision of Dr. R.P. Murrmann, Research Chemist, Earth Sciences Branch, Research Division, U.S. Army Cold Regions Research and Engineering Laboratory (USA CRREL) in association with C.I. Collins, Research Chemist, of the Mine Detection Division, U.S. Army Mobility Equipment Research and Development Center (USA MERDC). The work was supported by USA MERDC under DA Project 1G672712AJ22. The report was technically reviewed by T.D. Buzzell and D.C. Leggett, USA CRREL, and Dr. J.R. Gonano, USA MERDC.

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13. ABSTRACT Atmospheric samples were taken at the soil surface above field-emplaced M-15 and M-19 antitank mines and military explosive Composition-B. These samples were analyzed to determine if trace chemicals generated by the explosive material are detectable. Cyclohexanone was positively identified and is attributable to the explosive. Calculations were made, based on the amount of cyclohexanone found, to approximate its flux rate through the surface.		
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by

T.F. Jenkins, W.F. O'Reilly, R.P. Murrmann and C.I. Collins

INTRODUCTION

Research to determine the feasibility of using trace gas detection to locate emplaced military mines has been underway for some time. A substantial portion of this effort has been directed toward determining the signature characteristics of the major components of solid explosives.* Volatile trace impurities, which account for only a small part of the solid but which are prominent in the vapor, have been studied as well.^{2 9 12 15 17} Other investigations have focused on evaluating the current detection technology available for these various classes of components.^{14 15 16} While studies like these are both important and highly relevant to the development of trace gas detection systems, there is a conspicuous absence of information regarding the demonstrated ability to detect any vapor associated with an explosive in the atmosphere above an emplaced mine.

A recent study¹⁷ has demonstrated that cyclohexanone is an important trace component in vapor emitted by military grade Composition-B. It has also been shown⁹ that cyclohexanone is detectable outside of intact metallic and nonmetallic antitank mines charged with Composition-B which have been enclosed in a restricted volume. It is significant that while cyclohexanone is a common component of vapor from Composition-B, it is not a normal component of the atmosphere. Even if cyclohexanone is not a suitable signature chemical on which to base the development of a field-operational mine detection system, it would be significant to determine if cyclohexanone vapors, generated within the mine casing itself, can be detected at the soil surface above emplaced military mines. The ability to detect these vapors under differing environmental conditions would give credence to the concept of mine detection by trace gas analysis.

EXPERIMENTAL

Description of mine field

In conducting these experiments, a mine field was prepared at Fort Belvoir, Virginia, in the fall of 1972 (Fig. 1). Two types of antitank mines utilizing Composition-B as the explosive material were chosen for study: the M-15, a metallic mine (Fig. 2a), and the M-19, a plastic mine (Fig. 2b). The mines were emplaced by experienced personnel from USA MERDC and the field was secured by intrusion detection devices to ensure that no tampering with the mines or soils was possible. The mines used in the study were standard military mines with the detonators removed. Pertinent information regarding these mines is given in Table I.

* Refs. 1, 3, 5, 6, 7, 8, 11 and 13.



Figure 1. Mine field at Fort Belvoir, Virginia.

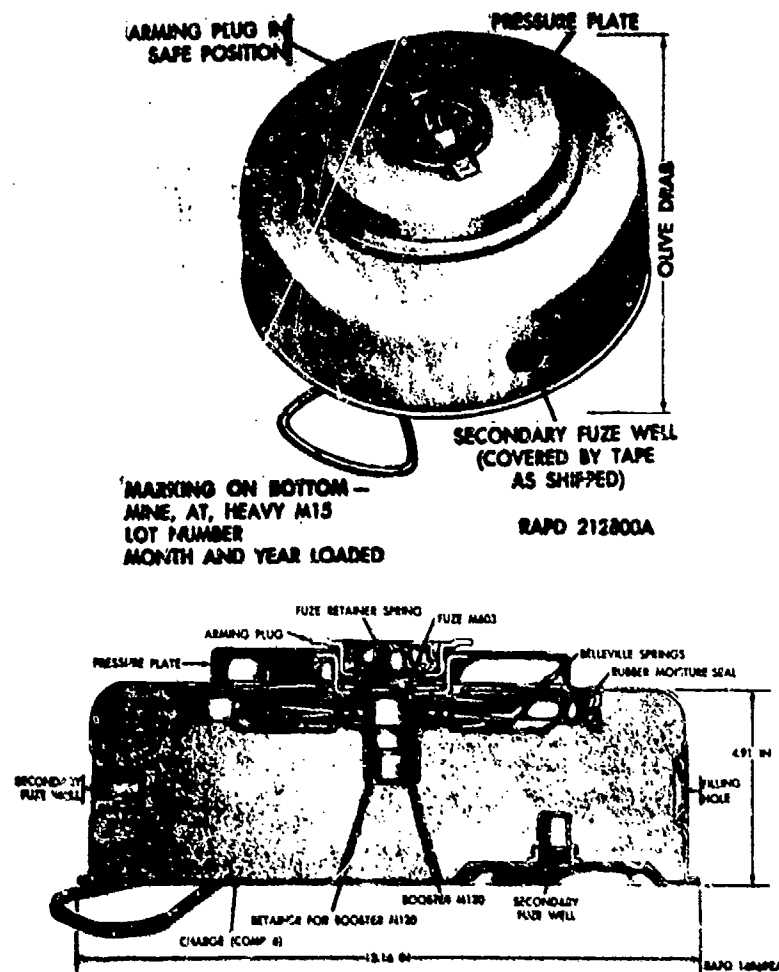
Table 1. Description of mines.

	M-15	M-19
Total weight (lb)	30	26
Weight of explosive (lb)	22	21
Type of explosive	Composition-B*	Composition-B*
Booster	None	Tetryl
Casing	Steel	Plastic
Lot number	LOP 14-14	LOP 500-1
Date of manufacture	1953	1967
Shape	Round	Box
Dimensions (in.)	4 $\frac{1}{2}$ × 13 $\frac{1}{2}$ diam	2.95 × 13.09 × 13.09

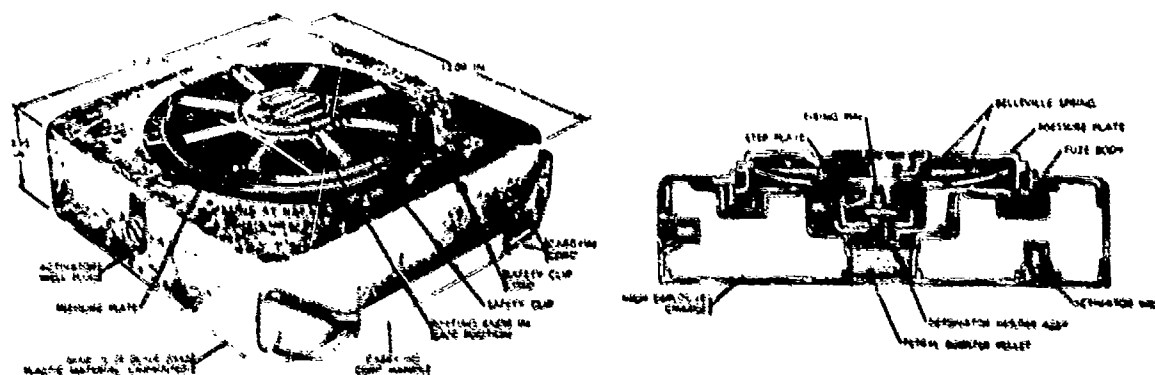
* Composition-B is composed of 40% TNT, 59% RDX and 1% wax.¹⁷

The initial configuration of the mine field as prepared in October 1972 is shown in Figure 3. In addition to the two types of mines, the following materials were also buried: Composition-B itself, sponges to which 20 ml of cyclohexanone had been added, inert mines (casing with no explosive), and mines in which cyclohexanone was added to the fuse well. The samples were emplaced with their top surfaces at depths of both $\frac{1}{4}$ and 2 in. (2 and 5 cm). The mine casing samples and empty holes, where soil was disturbed to the same two depths, were included to enable evaluation of natural background and contamination caused by handling or storage of samples. Cyclohexanone-spiked sponges were included as a high level source so that information regarding the permeability of the soil barrier to cyclohexanone vapor could be acquired quickly. In this initial configuration of the mine field, the soil used was that native to the site, Fort Belvoir clay.

In the final phase of the study, the mine field was reconfigured (Fig. 4). The sponges and mines with added cyclohexanone were eliminated in this second configuration. In addition to the Fort Belvoir clay, a second soil, a naturally occurring Windsor sand obtained near Hanover, N.H., was included. The physical and chemical properties of the two soils are summarized in Tables II and III. The soils represent the extremes in soil conditions normally encountered in the field. All the mines were reemplaced at a depth of 1 in., which was considered the minimum acceptable depth for a realistic evaluation.



a. M-15.



b. M-19.

Figure 2. M-15 and M-19 antitank mines. (From Department of the Army TM 9-1345-200.)

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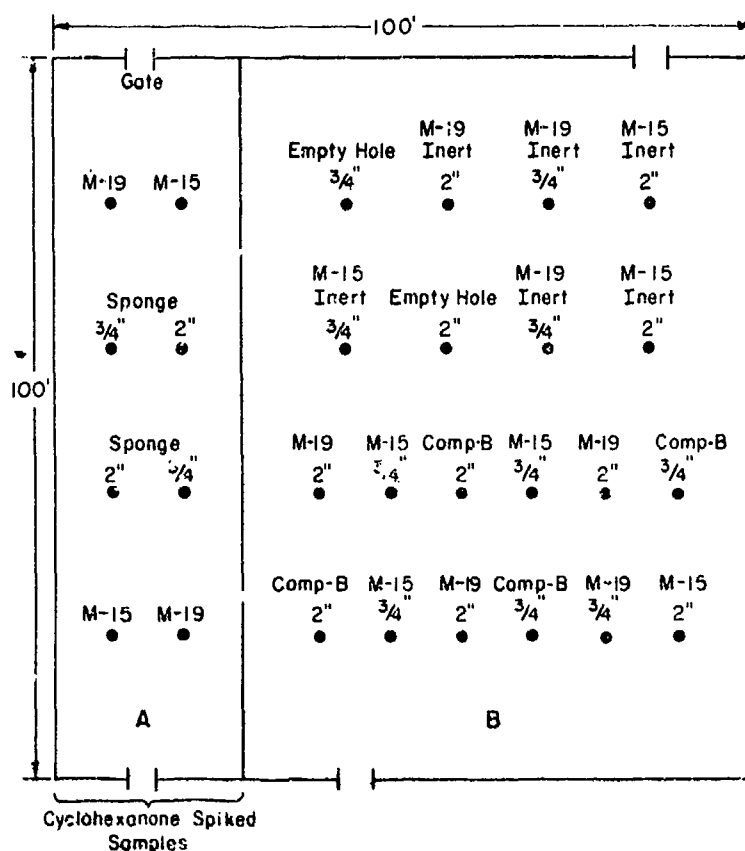


Figure 3. Preliminary mine field layout.

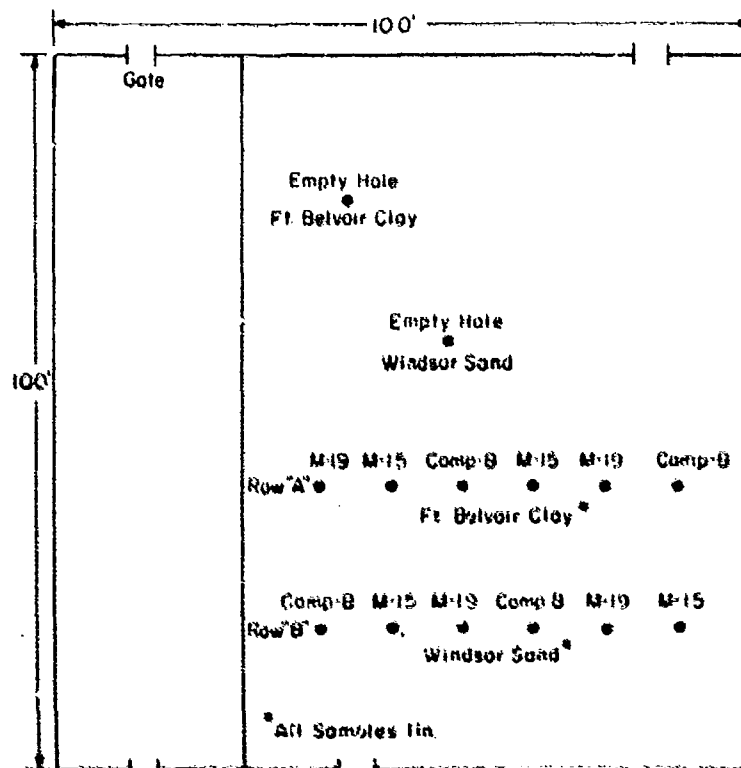


Figure 4. Final mine field layout (all samples at 1-in. depth).

Table II. Particle size analysis.

Diameter 0.074 mm or larger determined by sieve analysis; diameter less than 0.074 mm determined by hydrometer method.

Ft. Belvoir clay		Windsor sand	
Effective diam (mm)	Percent finer fraction (by weight)	Effective diam (mm)	Percent finer fraction (by weight)
25.4	100.0	25.4	100.0
9.52	89.2	0.84	86.7
2.00	83.6	0.42	62.3
0.84	80.8	0.210	33.6
0.42	76.5	0.149	29.1
0.210	67.2	0.074	18.0
0.149	66.1	0.0430	9.9
0.074	59.8	0.0321	6.9
0.0365	58.0	0.0236	4.6
0.0266	50.0	0.0170	3.2
0.0197	44.0	0.0126	2.3
0.0145	38.0	0.0090	1.9
0.0109	34.0	0.0064	1.3
0.0080	28.0	0.0046	0.76
0.0058	24.0	0.0032	0.38
0.0041	21.0	0.0025	0.38
0.0030	18.0	0.0013	0.38
0.0023	16.0		
0.0012	13.0		

Table III. Chemical properties of study soils.

Parameter	Fort Belvoir clay	Windsor sand
Total carbon (%)	0.30	0.62
Total phosphorus (ppm)	118.8	154.1
Extractable phosphorus (ppm)	7.0	35.6
Total nitrogen (%)	0.031	0.061
Free Fe ₂ O ₃ (ppm)	2.69	1.15
pH	5.2	6.2
Soluble salts (mmhos/cm)	0.20	0.35
Cation exchange capacity (meq/100 g)	7.7	3.1
Exchangeable cations (ppm)		
Ca	152.0	305.0
Mg	91.0	13.0
K	10.0	12.0
Na	15.3	11.0

Sample collection

In order to collect and concentrate vapors from the emplaced mines and explosives at the atmosphere/soil interface, a funnel-shaped sampler was prepared (Fig. 5). This device was 46 cm (18 in.) in diameter at the base by 20 cm (8 in.) high, and was made from 22-gauge stainless steel. It was equipped with a 0.32-cm (1/8-in.) swagelock connector at the top. The device was placed on the soil surface over an emplaced mine, thus restricting the atmospheric dispersion of mine vapors which had diffused through the soil barrier to the surface during the sample collection period. Samples collected during preliminary work indicated that contribution of vapors from the sampler itself was negligible.

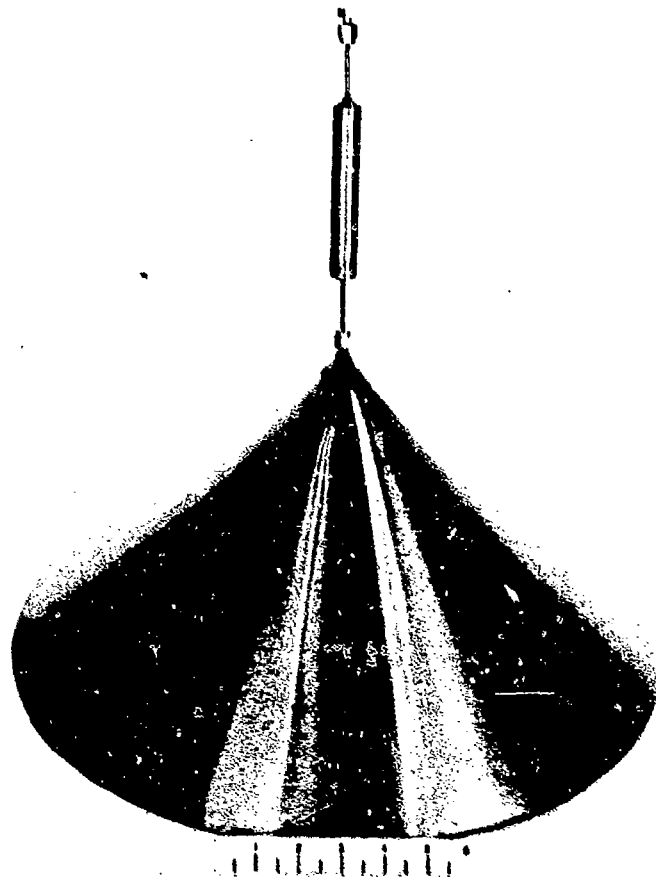


Figure 5. Funnel sampler with adsorption tube in place (scale in inches).

The actual collection and concentration of mine vapors was accomplished by drawing sample air at a predetermined rate through Chromosorb 102 adsorption tubes.^{1, 2} These tubes concentrate low-level organic components from large volumes of atmospheric air at ambient temperature without collecting substantial amounts of water. These same adsorption tubes were used successfully in a previous study³ to prepare samples of mine vapors for gas chromatographic analysis. During the exploratory phases of this investigation, several sample collection procedures, which differed in equilibration time, pumping rate and sampling time, were used. By trial and error the following optimized procedure evolved and was used for the final set of samples.

The sampler was positioned on the surface above the mine or explosive and vapors were allowed to equilibrate for approximately 17 hours. One end of a Chromosorb-102 collection tube was connected to the swagelock fitting on the funnel and the other end to a vacuum pump. Prior to the attachment of the vapor adsorption tube to the funnel sampler, the flow rate of the air through the tube was preset at 1 liter/min using a rotameter. The adsorption tube was then attached to the sampler, and the air above the mine was sampled for approximately seven hours. The sampler was then removed, allowed to "air out" for 30 minutes, and positioned over another mine overnight to allow the vapors to equilibrate the volume. In practice four samplers were operated simultaneously. Atmospheric air samples were taken through the sampler following routine sample collection to determine if any residual cyclohexanone remained on the funnel. None was found.

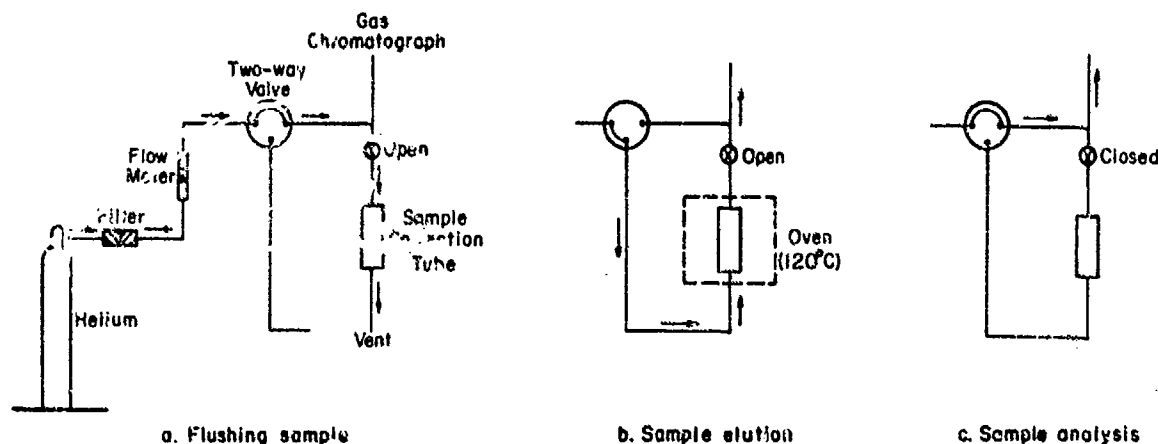


Figure 6. Inlet system for transfer of explosive vapors to gas chromatograph/mass spectrometer.

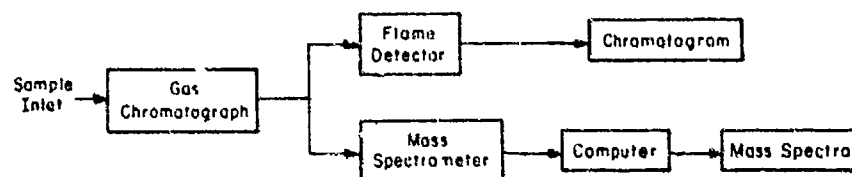


Figure 7. Experimental arrangement of gas chromatograph/mass spectrometer.

Analysis procedure

Analyses were performed on a Perkin-Elmer 270-B gas chromatograph/mass spectrometer (GC/MS). This instrument had been modified to incorporate a flame ionization detector and was connected on-line to a Digital Equipment Corporation PDP-12 computer for mass spectral data acquisition and analysis. Samples were analyzed using a 2.7 m \times 3.2 mm (9 ft \times $\frac{1}{8}$ in.) OD Durapak* column (Carbowax 400 chemically bound to Porasil C). The following analytical procedure was used.

A Chromosorb-102 adsorption tube was attached to the injection port of the GC/MS. The port had been previously modified as diagrammed in Figure 6 to allow for direct transfer of the vapors from the adsorption tubes. A block diagram of the GC/MS analytical system is shown in Figure 7. The tube was first flushed in the direction of sample collection with zero-grade helium (50 ml/min) for 1 hour to remove most of the residual water from it (Fig. 6a). The unattached end of the adsorption tube was then connected with Teflon tubing to a two-way valve on the helium supply. With the valve configured for helium flow to bypass the adsorption tube and enter directly into the GC/MS, the column was preconditioned at 150°C for 5 to 10 minutes. The oven was then cooled to -75°C and the two-way valve configured to put flow (50 ml/min) through the adsorption tube and into the cold GC column (Fig. 6b). The adsorption tube was then heated to 110°C for 2 hours with a modified tubular furnace, during which time trapped components in the tube were transferred to the head of the GC column. After the 2-hour transfer period, the furnace was removed and the flow reduced to 25 ml/min. The two-way valve was repositioned to again put flow directly into the GC column (Fig. 6c) and the column temperature was programmed to increase from -75°C to 150°C at a rate of 16°C/min.

* Waters Associates, Framingham, Massachusetts.

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The effluent from the column was split 50% to the flame ionization detector and 50% to the mass spectrometer. As peaks were observed on the flame ionization detector, mass spectra were obtained and stored on magnetic tape by the PDP-12 computer. Identification of mass spectra was made by comparison of the data obtained with catalogues of mass spectral data.

The column was baked out at 150° for 12 hours between samples, and blank runs were made prior to each analysis.

RESULTS AND DISCUSSION

Samples were taken at the mine field in its preliminary configuration (Fig. 3) and analyzed in December 1972. These samples included vapors collected for only 1 hour over the emplaced M-15 and M-19 antitank mines, Composition-B, inert mines, cyclohexanone-spiked sponges, cyclohexanone-spiked mines, and disturbed soil. From the results it was positively determined from mass spectra that cyclohexanone could be detected above the emplaced sponges and mines spiked with cyclohexanone; however, no positive mass spectral confirmation was obtained for the other samples. Chromatographic peaks were observed in the flame ionization detector trace at the correct retention time for cyclohexanone for all samples from both Composition-B and the two types of mines, but insufficient amounts were present to obtain mass spectra. It seems worthwhile mentioning that the odor of cyclohexanone could readily be sensed above the cyclohexanone sponges only several hours after they were emplaced. Although there was no odor, detectable cyclohexanone still evolved from the soil several months later. After sampling, soil was removed from around the sponges and it was determined that the largest amount of cyclohexanone was located in the soil zone directly below the sponge. This suggested that the cyclohexanone moved downward in the soil, probably reflecting its solubility in infiltrating water. The air temperature was cool during the period of sampling, with temperatures below freezing at night. In the mornings, the soil surface was frozen at the beginning of sampling periods.

It was decided after this initial study to test and optimize the sampling procedure for cyclohexanone detection. One experiment involved sampling directly over non-emplaced mines and Composition-B with the sampler and adsorption tubes to determine if the emission of cyclohexanone could be detected without the soil barrier. In this case, cyclohexanone was identified in the vapors from the M-15 mines and Composition-B. This experiment verified that cyclohexanone from intact mines could be detected using the sample collection apparatus. In the case of a buried mine, it was reasoned that detection of cyclohexanone would be more difficult due to the soil barrier. Consequently, the following experiment was run to determine the optimum sampling procedure for the detection of cyclohexanone at low levels.

Vapor samples were collected above buried Composition-B and emplaced cyclohexanone-spiked M-15 mines, with varying flow rates (0.1 and 1 liter/min), equilibration times before sampling began (0 and 17 hours), and sample collection times (1 and 7 hours). It was generally established that the faster flow rate combined with long equilibration and sampling times resulted in the highest amount of cyclohexanone observed.

M-15 and M-19 mines and Composition-B chips were then emplaced in the mine field in the second configuration as described previously. Two months later, in June 1973, the final set of vapor samples was collected above the emplaced mines and Composition-B. The results of this study reflect the improved sampling procedures. Another factor may have been more favorable climatic conditions, in that the ambient temperature was warmer than during the initial sampling period and there was no rainfall for at least a week prior to sampling. Typical examples of the chromatograms obtained are shown in Figures 8-11, where peak numbers refer to the compounds

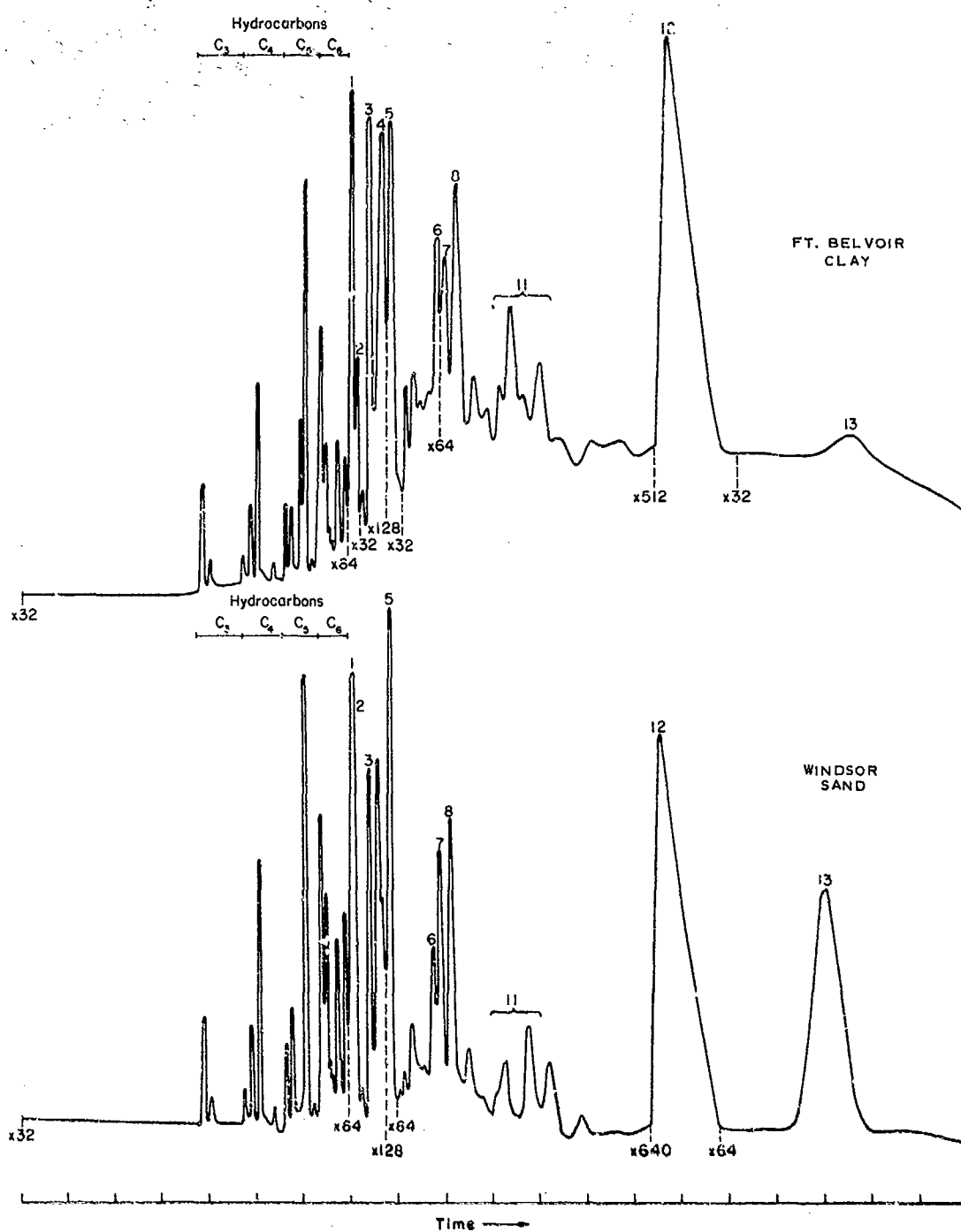


Figure 8. Chromatograms of samples taken over emplaced M-15 mines.

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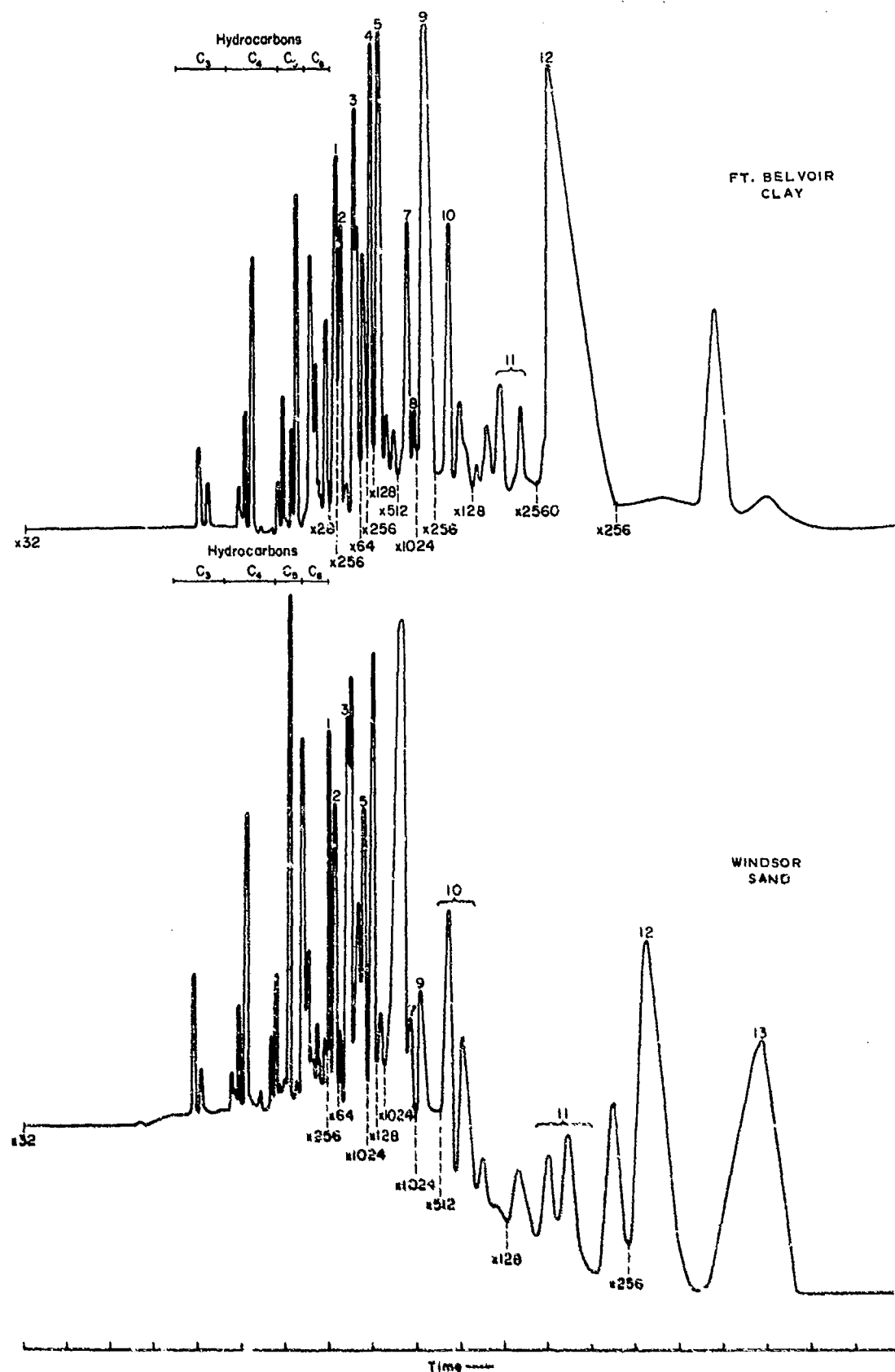


Figure 9. Chromatograms of samples taken over emplaced M-19 mines.

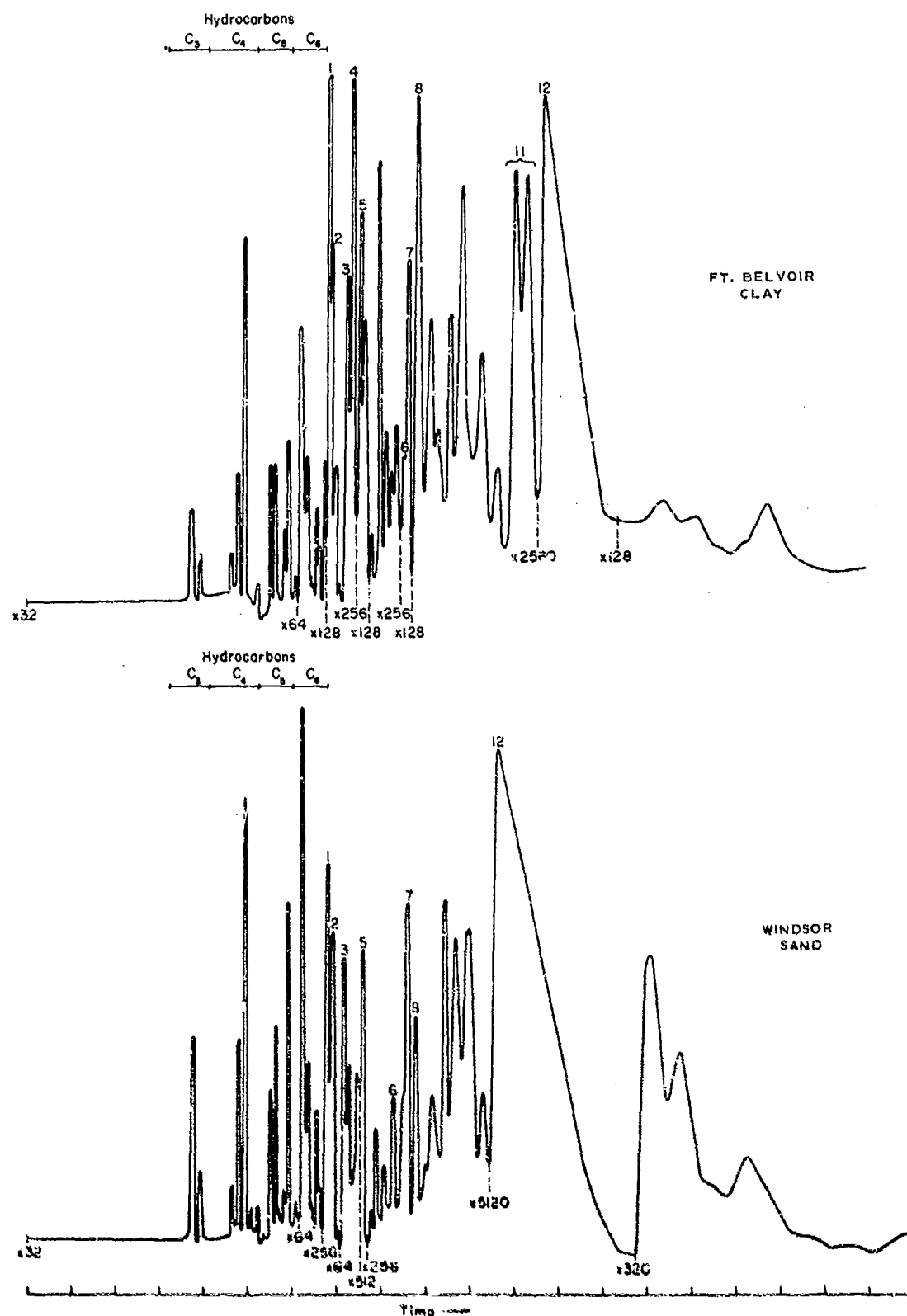


Figure 10. Chromatograms of samples taken over buried Composition-B.

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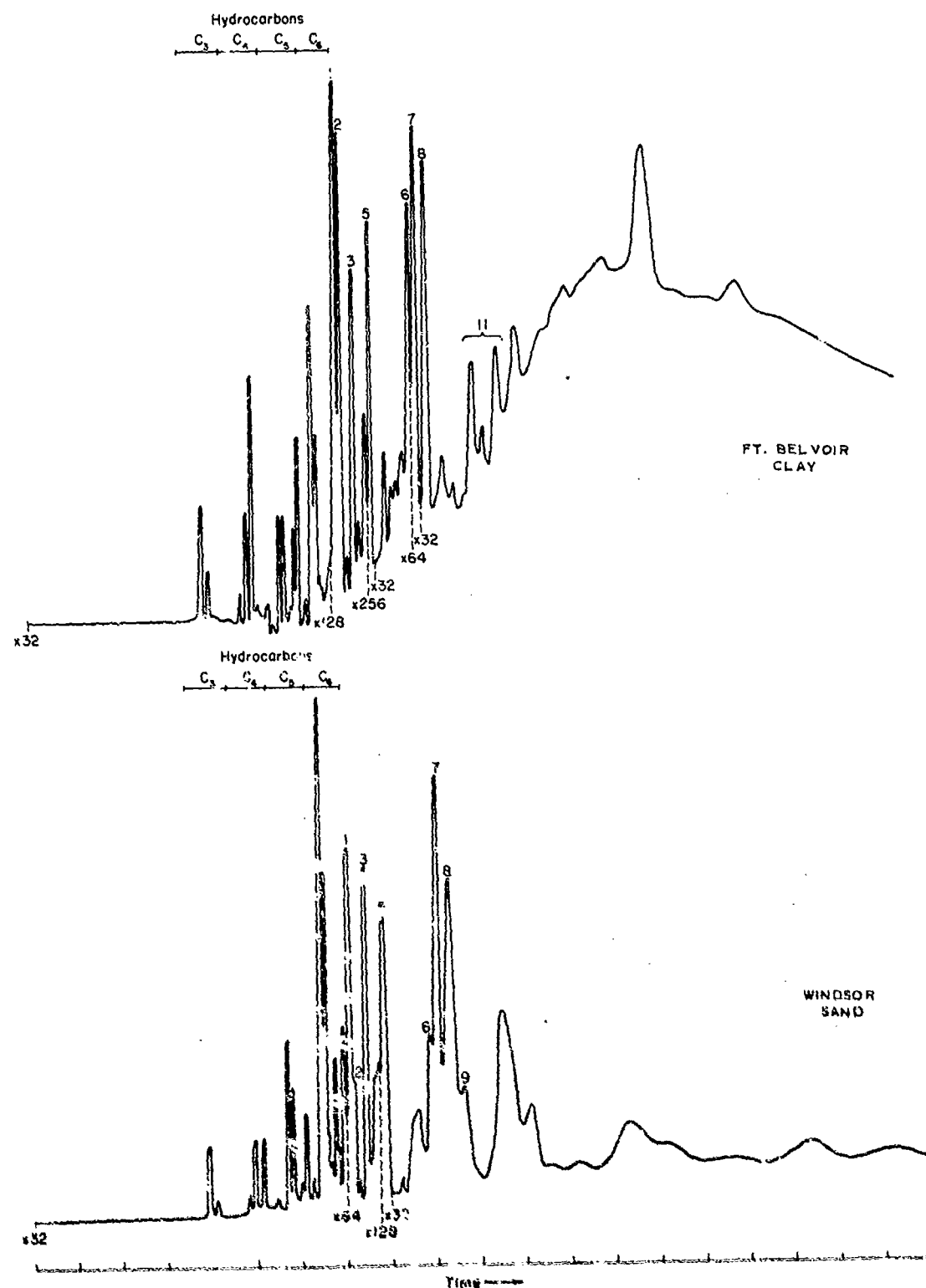


Figure 11. Chromatograms of samples taken over areas of disturbed soils.

Table IV. Compounds identified with the mass spectrometer for the chromatograms shown in Figures 8-11.

1. Benzene	8. Xylene
2. Trichloroethylene	9. Styrene
3. Tetrachloroethylene	10. C ₃ Aromatics
4. Acetone	11. C ₄ Aromatics
5. Toluene	12. Cyclohexanone
6. Ethylbenzene	13. Unknown (large peak at M/e = 281)
7. Xylene	

Table V. Cyclohexanone evolved from emplaced Composition-B charged mines and buried Composition-B.

Sample no.	Sample type	Soil type	Equilibration period (hr)	Sampling period (hr)	Cyclohexanone found ($g \times 10^3$)	Appearance rate ($g/sec \times 10^6$)	Flux ($g/cm^2 sec \times 10^{13}$)
1	M-15	Ft. Belvoir clay	17.0	6.5	4.8	5.7	6.5
2	M-15	Ft. Belvoir clay	16.5	7.0	2.8	3.3	3.8
3	M-19	Ft. Belvoir clay	17.0	6.5	11.8	14.0	13.0
4	M-19	Ft. Belvoir clay	16.5	7.0	14.0	16.5	15.0
5	Comp-B	Ft. Belvoir clay	16.0	7.5	14.2	16.6	15.0 [†]
6	M-15	Windsor sand	17.0	6.5	*	*	*
7	M-15	Windsor sand	16.5	7.0	2.8	3.3	3.8
8	M-19	Windsor sand	17.0	6.5	0.98	1.2	1.1
9	M-19	Windsor sand	16.5	7.0	8.8	10.4	9.4
10	Comp-B	Windsor sand	16.0	7.5	92.0	108.7	98.0 [†]
11	Comp-B	Windsor sand	16.5	7.0	42.0	49.6	45.0 [†]
12	Comp-B	Windsor sand	16.5	7.0	40.0	47.3	43.0 [†]

* Positive MS was not obtained although a small GC peak did appear at the correct retention time for cyclohexanone.

† A surface area equivalent to that for the M-19 mine was assumed to make this calculation.

listed in Table IV. The results of cyclohexanone detection are given in Table V. Cyclohexanone was unequivocally detected in 11 of the 12 samples taken from above the emplaced mines and Composition-B. In the one case where the presence of cyclohexanone could not be confirmed by mass spectra, a peak was observed on the flame ionization chromatogram with the correct retention time, but the concentration was below the detection limit of the mass spectrometer.

If an assumption is made that cyclohexanone appeared and was trapped at a constant rate during the time the soil was covered with the sampler, an appearance rate in g/sec can be calculated for each sample. These values (Table V) indicate that it should be possible to collect vapor for as little as a few seconds and have a sufficient amount of cyclohexanone for detection using a chromatographic technique with a flame ionization detector.

It is as yet unclear whether cyclohexanone is emitted from the mine in a small leak or over an extensive portion of the mine surface. If it is assumed, however, that cyclohexanone is evolving from the soil over an area roughly equivalent to the top surface area of the mine, a flux rate of

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cyclohexanone in g/cm² sec can be calculated. These values are given in the last column of Table V. A value for Composition-B is calculated using an area equivalent to the top surface area of the M-19 mine.

From the chromatograms given in Figures 8-11 it is clear that compounds other than cyclohexanone were also identified in these samples. From Figure 11, the samples taken over disturbed soils, it can be seen that the natural background, combined with a small number of peaks due to contamination of the adsorption tube collector, account for the majority of the other peaks shown in the chromatograms. None of the peaks, other than cyclohexanone, may be attributed to the mines or to Composition-B. The sampling and analysis procedures, however, were optimized for cyclohexanone detection, and as a result there was discrimination against many other types of compounds. In particular, if vapors of TNT or DNT¹⁵ evolved from the soil, these components would not elute as a peak from the analytical gas chromatographic column under the experimental conditions used. Modification of the collection and analysis procedure to determine the detectability of these vapors is currently in progress.

It is hoped that the values given in Table V will be of use to those interested in the concentration of vapors emitted from intact mines at the soil/atmosphere interface. Clearly the concentration of cyclohexanone which might be expected in the atmosphere above an emplaced mine is highly dependent on the meteorological conditions prevailing at that time; nevertheless, it is felt that this study has confirmed the feasibility of the concept of mine detection by trace gas analysis.

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